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RRM

AERO VAC CORP.

EFFECT OF LOW DENSITY INERT GAS FLUSH ON OXYGEN-COLUMBIUM REACTION AT 1500°F to 2000°F

Contract NASw-722

First Quarter Report

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INTRODUCTION

The gas purge process has shown its ability to achieve impurity partial pressures in the 10^{-6} to 10^{-7} torr range. The purpose of the program continuation is to extend the purity capability of the process. Such a program requires a decrease in the gas impurities, an increase in the detectability of the impurities and metallurgical proof that the purity has truly been extended. Below is the statement of work which the program will follow.

STATEMENT OF WORK

The investigation will include, but not necessarily be limited to, the following work:

(a) A study of the suitability of mass spectroscopy for analysis of oxygen and other impurities in argon or helium in the range below 1 part per million.

(b) Design and construction of a gas analyzer to perform the above named analyses.

(c) Use of the gas analyzer to continue the study of the practicability of obtaining reactive gas partial pressures in the 10^{-8} to 10^{-9} torr range by the inert gas flush system, including the effects of various system parameters.

(d) Continued comparison of the comparative utility and economy of the inert gas flush system and a static pressurized inert gas system.

At present, the mass spectrometer design for parts per billion impurity analysis is complete. Construction of the device is well underway. It is planned that purity analyses will begin before completion of the next quarter.

PROCESS APPLICATION

During the last quarter, several discussions with the people at CANEL (Pratt and Whitney) have prompted them to try the gas purge process. At present, they have many environmental chambers which are being used for testing liquid metal heat exchanger loops. The tests are performed essentially at atmospheric pressure, in an argon atmosphere. The argon is recirculated in a closed loop purifying system until a steady state purity of 1 - 25 ppm is attained. (At atmospheric pressure this corresponds to an impurity pressure of $7.6(10^{4})$ torr to $1.9(10^{-2})$ torr. At this point the test is begun. The loop is heated to approximately 2000°F. It has been determined experimentally that the gas purity is not adequate for long term tests and the columbium oxidizes quite badly.

A specific recommendation that they use the gas purge process was made.

The test is being performed on a reasonable size chamber (approximately 20 ft^{3}) with a mechanical pump and an argon supply purity of 1 ppm.

The important parameters were the connecting tube length and diameter. The tube diameter was taken as 2" and the tube length as 50" in the design calculations. The recommended flow rate was 0.4 torr-liters/sec. of 1 ppm gas with a pumping speed in excess of 10 liters/sec. This should result in impurity pressures due to the argon only of $4(10^{-8})$ torr. It was further recommended that the background pressure of the system be kept lower than the argon impurity pressure for optimum results. A good leak check and a 500°F degas to remove the water vapor are required procedures. The test is planned to run for 1000 hours. The results should be available towards the end of September.

MASS SPECTROMETER SYSTEM

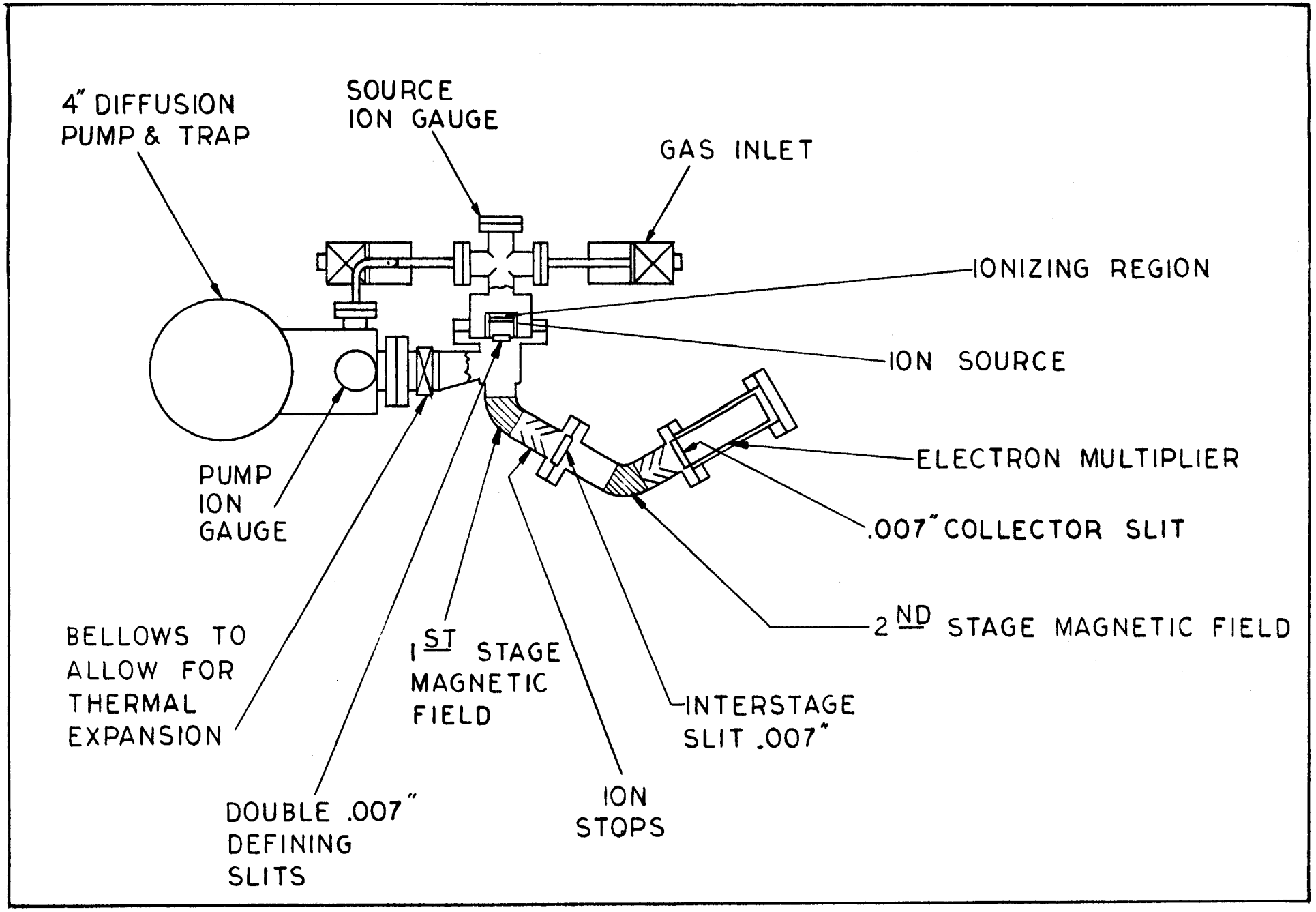
The design of the mass spectrometer system is shown on the attached layout.

Gas is sampled through the inlet valve from the higher pressure gas source (approximately .1 mm). The gas travels to the ion source region where it is ionized. The pressure in this region is maintained between 10^{-3} to 10^{-2} torr. This pressure is kept as high as this to obtain as many ions as possible. This high pressure, however, causes defocusing of the ion stream. However, this pressure is reduced by approximately 1000/1 at the other side of the defining slits. The pressure ratio is maintained by the high pumping speed to the 4" diffusion pump and the low conductance through the two series .007" thick defining slits. The ions are accelerated through the slits and must travel through a field-free region into the magnetic field. Any defocused ions are prevented from arriving at the interstage slits by the ion stops. Further elimination of defocused ions is achieved in the second stage where they pass through another magnetic field, before passing through the final collector slit. The ions then strike the 14 stage DuMont electron multiplier where the output is sensed.

TESTING PROGRAM

The results of the Phase I exposures are summarized in the table. Some of the data was reported in the final report of May 14, 1964. However, the data now is complete and is shown in its entirety. Two types of exposures were made, one in a static system and the other in the gas purge process at a pressure of .2 to .560 torr. The static tests are indicated in column (8) by the samples that are labeled "0" liters/sec pumping speed and column (9) by 1 atm. pressure. Column (6) gives the O_2 content of the sample at various sections. The first two (1 and 2) and the last two (7 and 8) sections were used to determine O_2 content. The middle four sections were used for the Knoop hardness test. Two interesting facts are borne out, the gettering rate increases with temperature, and in all cases results indicate equivalent impurity to that of a vacuum system at a pressure of 10^{-7} to 10^{-6} torr. This data was obtained from C. A. Barrett of NASA. This in general agrees with the impurity analysis of the gases made during the test (1-10 ppm). Operating total pressure was varied between .2 and .560 torr, corresponding to an impurity pressure of $2(10^{-7})$ as a minimum and $5(10^{-6})$ as a maximum impurity level.

Discussions with C.A. Barrett of NASA, Lewis Research Center, indicated a strong preference for several samples exposed to the purging atmosphere for a period of 1000 hours at 1850°F. Plans are presently being formulated to perform this experiment with the existing equipment.


4" DIFFUSION PUMP & TRAP
SOURCE ION GAUGE
GAS INLET
IONIZING REGION
ION SOURCE
ELECTRON MULTIPLIER
.007" COLLECTOR SLIT
2 ND STAGE MAGNETIC FIELD
INTERSTAGE SLIT .007"
ION STOPS
DOUBLE .007" DEFINING SLITS
1 ST STAGE MAGNETIC FIELD
BELLOWS TO ALLOW FOR THERMAL EXPANSION
PUMP ION GAUGE

TABLE

(1) Sample	(2) Temp. of Exposure (°F)	(3) Time of Exposure (hrs)	(4) Wgt. Gain (mg)	(5) Knoop Hardness Test	(6) O_2 Analysis (PPM) 1	2	7	8	(7) Pump Speed ℓ/sec.	(8) Pressure (Torr)
4	1500	239	0.0	108.6	830	810	810	820	2	.540
2	1500	23	- .08	120.1	1070	1070	740	740	2	.350
5	1500	24	.21	113.5	900	910	950	980	0	1 atm.
8	1750	164	.49	101.6	1070	1080	1050	1060	2	.410
3	1750	21.6	- .06	110.0	870	700	910	920	2	.540
6	1750	23.4	.38	112.2	1020	1010	1113	1118	0	1 atm.
13	2000	200	10.83	128.4	3600	2500	2600	2200	2	.560
9	2000	14	1.41	-----	1050	1060	1230	1400	2	.200
14	2000	26.8	.18	-----	3600	2500	2600	2200	0	1 atm.
Unexposed	0	0	-----	-----	950	950	950	950		
12			- .01	93.7	970	1060	1060	1080		

Hardness test was performed with a 100 gram load.